

Electrooxidation of alcohols at a nickel oxide/multi-walled carbon nanotube-modified glassy carbon electrode

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Abstract Electrocatalytic oxidation of methanol and some other primary alcohols on a glassy carbon electrode modified with multi-walled carbon nanotubes and nano-sized nickel oxide (GCE/MWNT/NiO) was investigated by cyclic voltammetry and chronoamperometry in alkaline medium. The results were compared with those obtained on a nickel oxide-modified glassy carbon electrode (GCE/NiO). Both the electrodes were conditioned by potential cycling in the range of 0.1–0.6 V versus Ag/AgCl in a 0.10 M NaOH solution. The effects of various parameters such as scan rate, alcohol concentration, thickness of NiO film, and real surface area of the modified electrodes were also investigated and compared. It was found that the GCE/MWNT/NiO-modified electrode possesses an improved electrochemical behavior over the GC/NiO-modified electrode for methanol oxidation.

Keywords Electrooxidation · GCE/NiO/MWNT · Electrocatalyst · Primary alcohols · Methanol

1 Introduction

In recent years, several studies on the electrocatalytic oxidation of alcohols have been carried, mainly because of their importance in fuel cell technologies [1–17]. Among different primary alcohols, methanol is the most intensively investigated, due to its availability, low price, and easy storage as a liquid. Ethanol also appears to be a good alternative for methanol because of low toxicity and availability. The electrode material is an important parameter which determines the electrooxidation overpotential of primary alcohols. In this connection, different electrode materials including Ag, Au, Cu, Ni, Pd, Pt, and Pt-binary electrodes and a number of carbon-supported electrodes have commonly been used for the electrochemical oxidation of methanol [11, 14, 15, 18–21].

On the other hand, modification of the electrode surface by suitable materials is a promising approach for preparing electrodes with improved electrocatalytic activity and minimizing electrooxidation overpotential of small alcohols. In this context, a wide variety of compounds have been proposed as electrocatalyst for electrooxidation of alcohols. Salens, salophens, and anthrones coordinated to Mn, V, Fe, Co, Ni, Cu, Mo, Pd, and Sn [3, 22], a mixture of platinum tetraammine complex with cobalt quinolyldiamine complex [23], a series of single transition metalloporphyrins (V, Mn, Fe, Co, Ni, Cu, and Zn) and combination of two transition metalloporphyrins (V/Fe, Co/Fe, Ni/Fe, and Cu/Fe) [24], platinum microparticles dispersed on/in polypyrrole film [25], nickel macrocyclic complex-based films in alkaline aqueous solution [26–29], nickel(II) hydrotalcitetype anionic clay on a glassy carbon electrode [30] have been applied to the electrooxidation of methanol.

Carbon nanotubes are important class of materials due to their unique chemical, electrical, and structural characteristics

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[31]. Recent studies have demonstrated the ability of multi-wall carbon nanotubes (MWCNTs) in improving electrocatalytic properties of the modified electrodes [32, 33]. Several applications of carbon nanotubes as promising catalyst supports for fuel cells have recently been reported [34–41]. Considering the importance of nickel-based materials as suitable catalysts for the oxidation of alcohols, in addition to the spatial properties of CNTs, the target of the present work was the application of MWCNTs, as catalyst supports, to improve the electrocatalytic activity of glassy carbon/nickel oxide electrode in alkaline solution.

2 Experimental

2.1 Materials

Analytical grade methanol, ethanol, 1-propanol, 1-butanol, 1-pentanol, and NiNO_3 (all from Merck) were used as received. The concentrations of alcohols were adjusted by injection with a syringe of the required volume of alcohol in 0.1 M NaOH solutions. The MWCNTs with an O.D. \times I.D. of 40–70 nm \times 5–40 nm and height of 0.5–2 μm and >95 % purity were obtained from Aldrich and used without any further purification. All aqueous solutions were prepared using doubly distilled water.

2.2 Apparatus

All electrochemical measurements were conducted using an Autolab PGSTAT 30 potentiostat/galvanostat controlled by computer and GPIB card. The measurements were carried out using a conventional three-electrode cell using a Ag/AgCl, KCl (sat) as reference electrode; a Pt wire as the counter electrode; and a glassy carbon disk electrode (GCE, with a 0.0314 cm^2 geometrical area from Metrohm), in the form of bare or modified with NiO/MWCNTs, as working electrode. All experiments were carried out at room temperature. The surface of bare and modified glassy carbon electrodes were characterized by a Phillips Scanning Electron Microscope (SEM).

2.3 Modification of electrodes

The GCE was polished to a mirror finish with alumina slurry (0.3 and 0.05 μm), dipped in acetone for about 1 min in an ultrasonic bath, then washed with distilled water and ethanol for a few minutes. 1 mg of MWNT was dispersed in 10 mL *N,N*-dimethylformamide (DMF) with the aid of ultrasonic agitation to give a dark solution. 20 μL of the MWCNT solution was cast on the GCE surface and its solvent was evaporated at 50 $^\circ\text{C}$ to prepare the GCE/MWCNT-modified electrode. Modification of the electrode

was accomplished by transferring one drop of aqueous 2 mM $\text{Ni}(\text{NO}_3)_2$ solution to the GCE/MWCNT electrode surface and allowing it to dry at 50 $^\circ\text{C}$ in an oven for 25 min. The GCE/MWNT/NiO was finally prepared by 30 repeated voltammetric cycles between 0.1 and 0.6 V versus Ag/AgCl in 0.1 M NaOH solution as supporting electrolyte until a steady-state voltammogram was obtained. In addition, a nickel(II) oxide glassy carbon-modified electrode (GCE/NiO) was prepared similar to the case of GCE/MWCNT/NiO. This electrode was prepared to compare its behavior with that of the GCE/MWNT/NiO electrode. Different loadings of the modifying layer were obtained by varying the concentration of $\text{Ni}(\text{NO}_3)_2$ solution.

3 Results and discussion

3.1 GCE/NiO electrode

The electrochemical behaviors of bare GCE and GCE/NiO electrodes in 0.1 M NaOH are compared in Fig. 1. As is obvious, the bare GCE in 0.1 M NaOH solution showed no measurable response in the absence (Fig. 1a) and presence of methanol (Fig. 1b). However, the modified GCE/NiO in 0.1 M NaOH solution (Fig. 1c) revealed pronounced redox peaks, similar to those previously reported in the literature [27, 42–44]. It is seen that the redox peaks corresponding to Ni(II)/Ni(III) couple appeared at about 0.44 and 0.37 V versus Ag/AgCl, at a scan rate of 50 mV s^{-1} . It should be noted that an increase in scan rate resulted in increasing the peak to peak separation, in which the cathodic peak shifted toward more negative and the anodic toward more positive values. Accordingly, the electrochemical behavior of the GCE/NiO electrode markedly deviated from a surface-confined process with fast electron-transfer behavior [45, 46].

Typical cyclic voltammogram of the GCE/NiO electrode in alkaline solution containing 0.10 M methanol at a scan rate of 50 mV s^{-1} is shown in Fig. 1d. As seen, a new peak appeared in anodic scan at about 0.64 V versus Ag/AgCl, while no measurable peak was observed for methanol oxidation at the bare GCE (Fig. 1b). Thus, the appearance of this new anodic peak can be attributed to the methanol oxidation that takes place after the complete oxidation of $\text{Ni}(\text{OH})_2$ to NiOOH [46–49]. The height of peak at 0.64 V was found to increase with increasing methanol concentration while the peak located at 0.44 V, which is due to formation of Ni(III), remained almost unaffected in the presence of methanol. Similar behaviors have already been reported for ethanol and methanol oxidation at a GCE/NiO electrode [27, 42].

Figure 2a, b show the SEM patterns of bare GCE and GCE/NiO electrodes, respectively. It can be found that, although most surface area of GCE/NiO electrode is

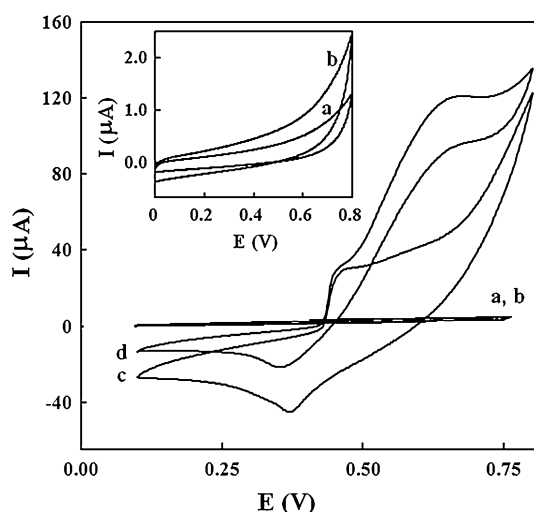


Fig. 1 Cyclic voltammograms obtained at a scan rate of 50 mV s^{-1} for bare GCE in 0.10 M NaOH in the absence (a) and presence of 0.10 M methanol (b) and for modified electrode GCE/NiO in 0.10 M NaOH in the absence (c) and presence of 0.10 M methanol (d). Inset shows the magnification of voltammograms a and b

covered by relatively uniform dispersed small-sized NiO spherical particles, there are some NiO agglomerates of larger size present at the surface which can induce some roughness at the modified electrode surface. As expected, at higher surface coverage, more and more NiO particles may join together so that the resulting increased agglomerate numbers can decrease in the effective area of the electrode surface, which will result in decreased catalytic efficiency of the electrode.

3.2 GCE/MWNT/NiO electrode

Figure 3a shows the cyclic voltammograms of modified GCE/MWCNT/NiO electrode in 0.1 M NaOH solution at various scan rates. Although the cathodic and anodic peak potentials were the same as that for a GCE/NiO electrode (Fig. 1), the cathodic and anodic peak currents have been increased significantly. The increased peak current can be attributed to the larger surface area of the GCE/MWNT/NiO-modified electrode.

In Fig. 2c, d the SEM patterns of GCE/MWCNT- and GCE/MWCNT/NiO-modified electrodes, respectively, are shown. As is seen from Fig. 2c, at the surface of the GC electrode, bundles of MWCNTs, with a general diameter in the range 40–70 nm, are crosslinked with each other to form a highly porous platform suitable for the immobilization of NiO nanoparticles. Meanwhile, Fig. 2d indicates a nice homogeneous coating of the NiO nanoparticles (with an average size of about 230 nm) in the highly porous surface of GCE/MWCNTs electrode, after application of

30 repeated voltammetric cycles to the added $\text{Ni}(\text{NO}_3)_2$ solution between 0.1 and 0.6 V versus Ag/AgCl in 0.1 M NaOH solution as supporting electrolyte. The SEM image illustrates the randomly deposited nano-sized NiO assemblies at the electrode surface, which clearly suggesting the three-dimensional dispersion of the catalytic centers. It can be seen that the vacant spaces still exist between the strongly adsorbed NiO assemblies, which are necessary for the access of substrate to the catalytic sites.

Figure 3b shows the linear dependence of the anodic peak current on the scan rate for GCE/MWCNT/NiO electrode in 0.1 M NaOH, which is a normal behavior expected for a surface-adsorbed substance. The observed increase in the peak separation with increasing scan rate and the independence of $I_{\text{pa}}/v^{1/2}$ on v (Fig. 3c) corresponds to a diffusion-controlled quasi-reversible process.

3.3 Oxidation of alcohols at GCE/MWCNTs/NiO-modified electrode

The electrocatalytic behavior of the modified GC/MWCNT/NiO electrode toward the electrooxidation of methanol, ethanol, 1-propanol, 1-butanol, and 1-pentanol in alkaline aqueous solution was investigated by cyclic voltammetry. The results obtained clearly revealed that the modified electrode acts as an excellent catalyst for the electrochemical oxidation of alcohols in 0.1 M NaOH aqueous solution. The cyclic voltammograms of the GCE/MWCNT/NiO electrode in a 0.1 M NaOH aqueous solution in the absence (scan a) and presence of 0.1 M methanol solution (scan b) are shown in Fig. 4a. Noteworthy, Fig. 5 shows that, among the small primary alcohols studied, the electrochemical behavior is more or less similar. Thus, methanol was investigated in more detail as a model molecule. In contrast to the redox activity observed at the GCE/NiO electrode around 0.6 V (Fig. 1), the GCE/MWCNT/NiO electrode exhibited considerable oxidation currents over the same potential range, as illustrated in Fig. 4b. Additionally, the efficiency of the prepared GCE/MWCNT/NiO electrode for electrooxidation of the mentioned alcohols, distinguished by anodic peaks current density, is significantly high in comparison with many other modified electrodes reported in the literature [2, 6, 21, 24–26, 44, 50–54].

It should be noted that a considerable increase in the currents for the case of electrooxidation of alcohols indicates the important role of MWCNTs in these reactions. The porous structure and small dimension of MWCNTs provided a larger surface area of the modified electrode and, consequently, enhanced its adsorptive performance for alcohols and resulted in significant increase in the corresponding peak currents.

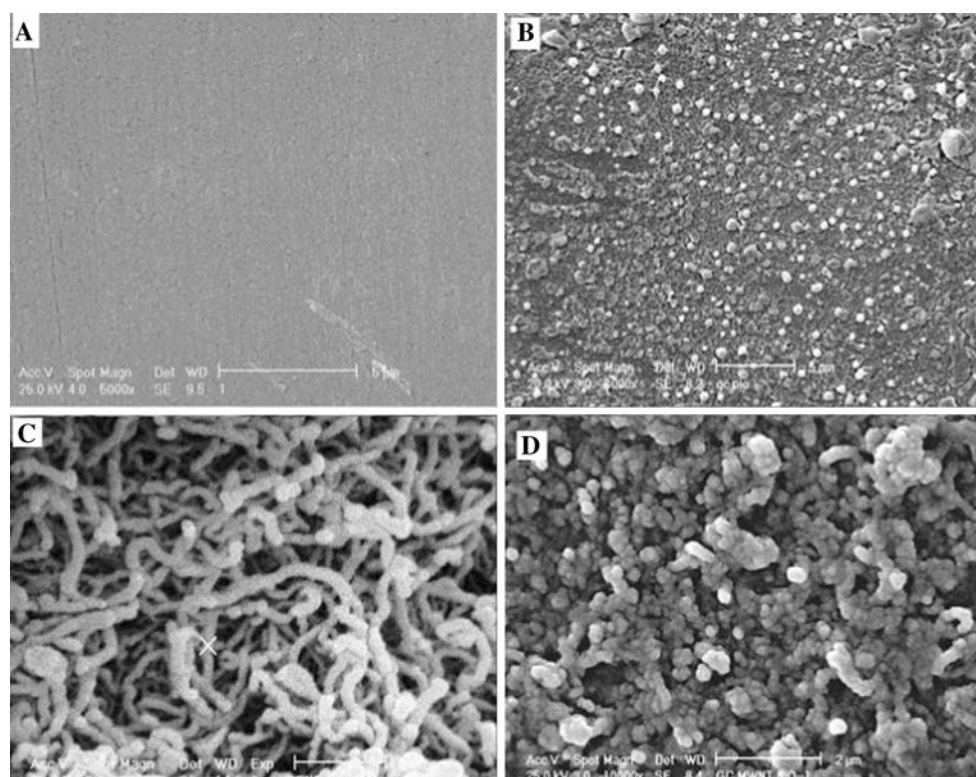


Fig. 2 SEM images of bare GCE (a), GCE/NiO (b), GCE/MWCNT (c), and GCE/MWCNT/NiO (d) modified electrodes

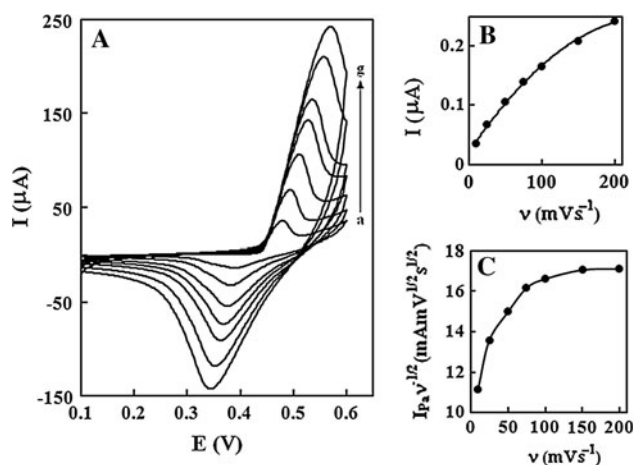


Fig. 3 **a** Effect of scan rate on the voltammograms of GCE/MWCNT/NiO-modified electrode, recorded in 0.1 M NaOH and scan rates of 10 (a), 25 (b), 50 (c), 75 (d), 100 (e), 150 (f), and 200 (g) mV s^{-1} . **b** Plot of anodic peak currents versus scan rate. **c** Plot of $I_{pa}/\nu^{1/2}$ versus scan rate

3.4 Effective parameters on the oxidation of alcohols at GCE/MWCNT/NiO-modified electrode

3.4.1 Influence of scan rate

The effect of scan rate on the catalytic electrooxidation of alcohols at GCE/MWCNT/NiO and GCE/NiO electrodes

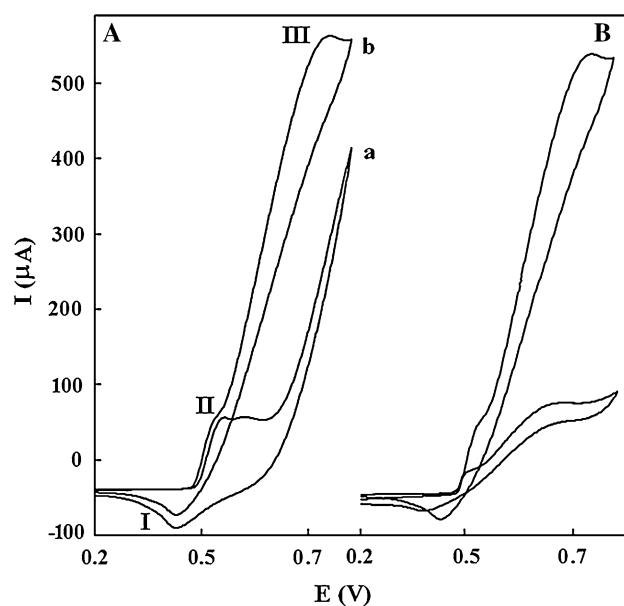


Fig. 4 **a** Cyclic voltammograms of GCE/MWCNT/NiO-modified electrode in the absence (a) and presence of 0.10 M methanol in 0.10 M NaOH (b). **b** Comparison between CV for oxidation of methanol on GCE/NiO electrode (lower CV, taken from Fig. 1b) and that on GCE/MWCNT/NiO (upper CV, taken from Fig. 4A, b)

was investigated by cyclic voltammetry. Figure 6a, b shows the dependence of the anodic to cathodic peak currents ratio (I_{pIII}/I_{pI}) on the scan rate for the

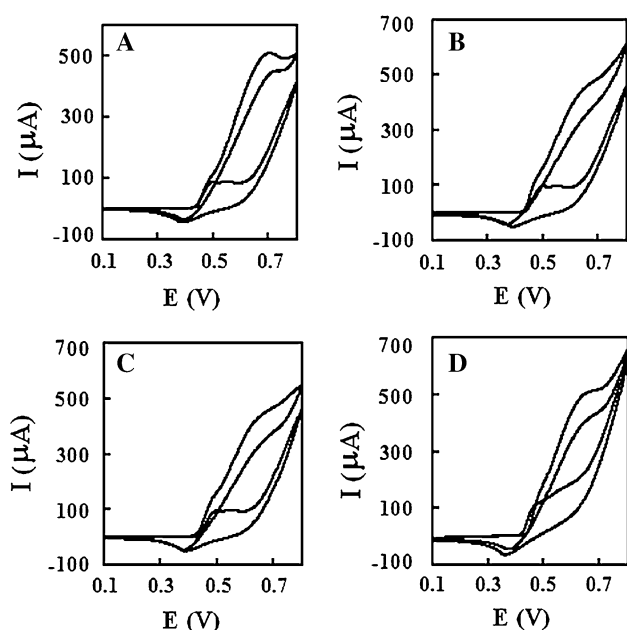


Fig. 5 Cyclic voltammograms of GCE/MWCNT/NiO-modified electrode in 0.10 M NaOH in the absence (lower CV) and presence (upper CV) of 0.10 M of ethanol (a), 1-propanol (b), 1-butanol (c), and 1-pentanol (d)

GCE/MWCNT/NiO and GCE/NiO electrodes in 0.1 M of CH_3OH in 0.1 M NaOH, respectively. The I_{pI} , I_{pII} , and I_{pIII} have been marked in Fig. 4a. As it is seen, the peak current ratios ($I_{\text{pIII}}/I_{\text{pI}}$) and ($I_{\text{pIII}}/I_{\text{pII}}$) decrease with increasing scan rate for both electrodes. Alternatively, the corresponding peak currents for the oxidation of NiO (I_{pII}) and the reduction of NiOOH (I_{pI}) increase with increasing scan rate. Hence, it can be concluded that the NiO/NiOOH transformation process is much faster than that of methanol oxidation and the catalytic reaction between methanol and NiOOH is a slow process. However, increase in the peak currents for the GCE/MWCNT/NiO electrode is higher than that for GCE/NiO electrode, because of the effective role of the MWCNTs. The stability of the GCE/MWCNT/NiO electrode was tested. After the modified electrode was dried and stored in air for two weeks, the intensity of its CV peaks showed only a slight decrease of <5 %.

3.4.2 Effect of methanol concentration

Electrooxidation currents for alcohols increase proportional to their concentrations in the solution. Figure 7 shows the effect of methanol concentration ranging from 0 to 0.25 M on the cyclic voltammograms of GCE/MWCNT/NiO and GCE/NiO electrodes in 0.1 M NaOH at the scan rate 50 mV s^{-1} . It is seen that, in both cases, the peak currents increase with increasing methanol concentration until a concentration of 0.15 M is reached and then they will stay almost constant and independent of concentration. Thus, a

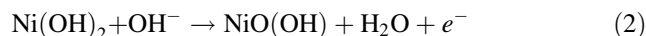
concentration value of 0.15 M represents a critical concentration, at which the active sites of the electrode surface will be saturated completely. In addition, it is clearly observed that modified electrode with MWCNTs show higher peak currents.

3.4.3 Effect of deposited amounts of nickel on electrooxidation of alcohols

The dependence of methanol oxidation current peak on the loading of Ni(II) species in the GCE/NiO and GCE/MWCNT/NiO electrodes were investigated. The results obtained revealed an increasing dependence on the Ni(III) surface concentration for both electrodes, up to surface concentrations of $3.5\text{--}4.0 \text{ mol cm}^{-2}$ (Fig. 8). The surface concentrations were calculated according to the following equation:

$$\Gamma = \frac{Q}{nFA} \quad (1)$$

where Γ is the surface concentration in mol cm^{-2} , Q is the charge under the oxidation peak for the voltammogram recorded in 0.1 M NaOH, A is the geometric surface area of the glassy carbon electrode, and n is the number of electrons transferred in the reaction:



However, at higher films thickness, the peak currents decreased most probably due to the decreased film porosity as a result of the formation of compact layers [53]. In addition, a thicker film leads to some difficulties in electron transfer through the film itself.

3.4.4 Effect of the real surface of electrode

The effect of the real surface area of the modified electrodes, as an effective parameter in determining the peak currents, in the catalytic reaction were investigated by chronoamperometry, and the resulting $I-t^{-1/2}$ plots obtained for GCE/NiO and GCE/MWCNT/NiO electrodes in solutions containing 0.10 M methanol and 0.10 M NaOH are shown in Fig. 9.

It should be noted that the corresponding chronoamperograms were recorded by setting the electrode potentials at 0.8 V (vs Ag/AgCl). As seen, the $I-t^{-1/2}$ plots give a straight line under diffusion control according to the Cottrell equation [54]:

$$I = \frac{nFA_{\text{real}}D^{1/2}C^*}{\pi^{1/2}t^{1/2}} \quad (3)$$

where D is the diffusion coefficient and C^* is the bulk concentration of methanol. The above-mentioned equation can be used to estimate the real surface area (A_{real}) of

Fig. 6 **a** Variations of I_{pIII}/I_{pI} (a) and I_{pIII}/I_{pII} (b) ratios with scan rate in the presence of 0.10 M methanol for GCE/MWCNT/NiO. **b** Same plots as **a** for GCE/NiO-modified electrode

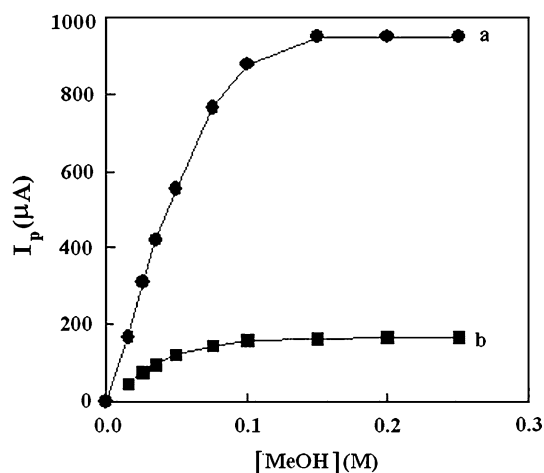
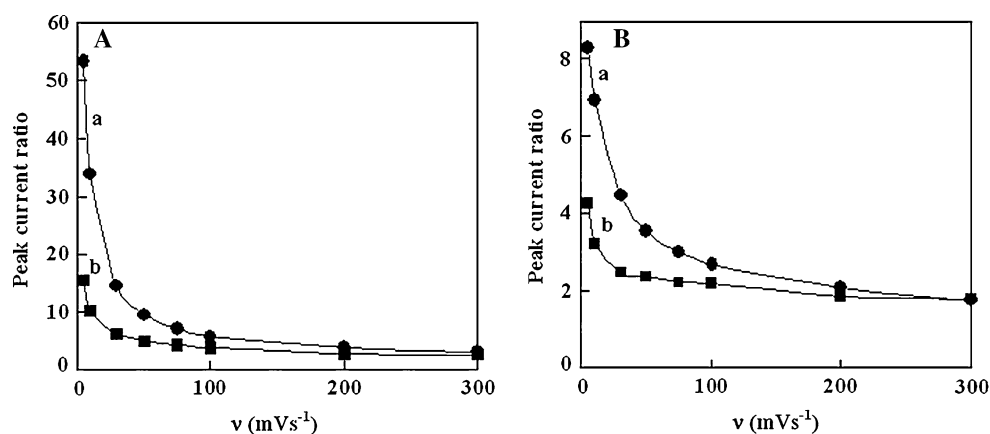


Fig. 7 Variation of the peak current for methanol oxidation at GCE/MWCNT/NiO (a) and GCE/NiO (b) modified electrodes with methanol concentration

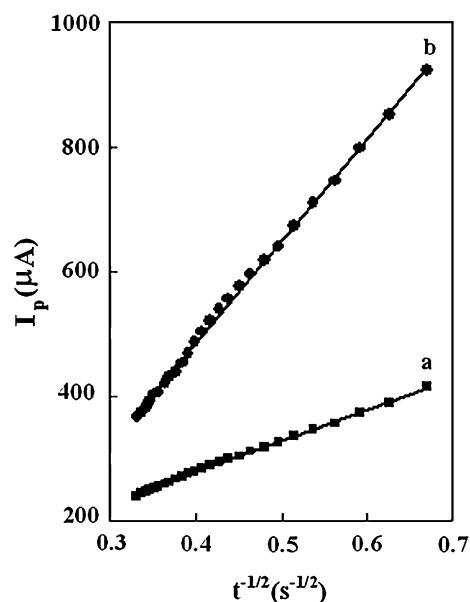


Fig. 9 Plots of currents versus $t^{-1/2}$ derived from chronoamperograms, at a potential step was 0.8 V versus Ag/AgCl, for GCE/NiO (a) and GCE/MWCNT/NiO (b) electrodes in 0.10 M of methanol in 0.10 M NaOH

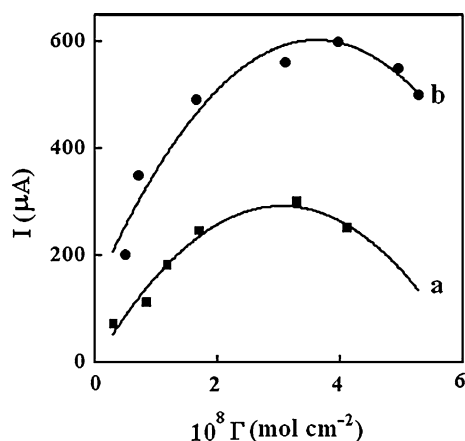


Fig. 8 The dependence of methanol oxidation current peak on the surface concentration of NiO species at the GCE/NiO (a) and GCE/MWCNT/NiO (b) electrodes in 0.10 M methanol and 0.10 M NaOH at a scan rate of 50 mV s^{-1}

electrodes. Comparing the slopes of these plots revealed that the real surface area of the GCE/MWCNT/NiO

electrode is 3.26 times higher than that of the GCE/NiO electrode. This can clearly explain the critical role of MWCNTs in amplification of the electrocatalytic currents.

4 Conclusions

Comparison of electrocatalytic behavior of the GCE/NiO and GCE/MWCNT/NiO electrode toward some primary alcohols in alkaline solution revealed that the modified electrode with MWCNTs and NiOOH formed during the oxidation of NiO in alkaline medium improves markedly the electrocatalytic behavior. The GCE/MWCNT/NiO electrode was found to be stable even under dry conditions for more than 2 weeks. In addition, the efficiency of the

GCE/MWCNT/NiO electrode toward the electrooxidation of alcohols which distinguished by the anodic peaks current density is significantly high in comparison with many of other previously reported modified electrodes.

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